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DEVELOPMENT AND TRIAL FIELD APPLICATION OF A QUALITY ASSURANCE PROGRAM FOR DEMONSTRATION PROJECTS

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1.0 INTRODUCTION

The major objective of this project was to develop a general quality assurance (QA) program for EPA demonstration projects, using the wet limestone scrubber facility at the Shawnee steam plant, Paducah, Kentucky, as an example project. A second objective was to field test the QA program at the Shawnee facility and carry out whatever modifications were necessary in light of that field trial.

Two concurrent final reports deal separately with these objectives and should be consulted for further treatment of areas of interest mentioned here. These reports are:

1. Guidelines for Demonstration Project Quality Assurance Programs, EPA-600/2-76-081.
2. A Quality Assurance Program for the Environmental Protection Agency Wet Limestone Scrubber Demonstration Project, Shawnee Steam-Electric Plant, Paducah, Kentucky, EPA-600/2-76-080.

Reference will occasionally be made to these reports. For conciseness, reports 1. and 2. will be referred to as the guidelines report and the Shawnee report, respectively.

Organization of this report is as follows: section 2.0 discusses significant areas for quality control (QC) from the RFP to the day-to-day QC program; section 3.0 outlines a quality assurance program for demonstration projects; section 4.0 treats the specific project studied, the wet limestone scrubber at the Shawnee steam plant; and section 5.0 is an evaluation of the QA program implemented at Shawnee. The major areas investigated at Shawnee were the analytical (control) laboratory, the gas stream measurement systems, and the process monitoring and control instrumentation.

To facilitate the reading of this report, two terms should be carefully defined. These terms are quality control and quality assurance. Concise definitions are given herewith.

Quality control: the overall system of activities the purpose of which is to provide a quality of product or service that meets the needs of users.

Quality assurance: A system of activities the purpose of which is to provide assurance that the overall quality control job is being done effectively.

Recommendation of a quality control system as such does not fall within the scope of this project. It is important, however, to be aware of the elements of such a system if one is to act as a monitor of QC by means of QA work. For this reason, the first major area addressed by this report is the QC system itself.

2.0 MAJOR COMPONENTS OF A QUALITY CONTROL PROGRAM

2.1 Quality Assurance Aspects of the RFP

The design of the RFP is predicated on stating as clearly as possible what the objectives of the project are; e.g., to design, construct, and maintain a given control system, systematically examining the interaction of appropriate system parameters. The quality of the data obtained from the project will depend upon numerous factors--instrumentation, personnel, sampling technique, sampling size, statistical expertise. It is therefore critical that the RFP be as explicit as possible* in delineating two things--what quality data are expected, and how that quality is to be insured.

Since most RFP's are limited in length, it would usually be inappropriate to include more than a brief (one- or two-paragraph) statement of QC requirements. Nevertheless, it is most important that the bid solicitation be as explicit as possible concerning QC.

2.2 Evaluation of Quality Control in the Proposal

The proposal should contain a statement as to the precise position the bidder's company takes regarding quality control programs. This should include past projects and the quality control program effectiveness in that project. In particular, there should be a clear and explicit response to the QC requirements stated in the RFP. This response must be compared directly, item-by-item, with other proposals submitted against the RFP. The evaluation should result in a determination of a "figure of merit" for the bidder's quality control organization and the competence of the staff.

If a contractor has a good proposal but is unclear on some phases of data quality, it would seem worthwhile to have him clarify his proposal by asking him to answer specific questions. If the answers to these questions are still vague, it is a good indication that the quality for these phases of the project may be questionable if this contractor carries out the project.

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*It is understood that, because of the nature of the proposed work, it may not be possible to specify either the expected data quality or the way in which data quality is to be insured.

2.3 Evaluation of Quality Control in the Work Plan

The work plan should be a detailed accounting of the actual steps to be taken to complete the work delineated in the proposal and should be in direct accord with the requirements of the RFP and other agreements with the project officer. Particular attention should be placed on mutually agreed upon critical areas in order to realize the collection of data having acceptable precision, accuracy, representativeness, and completeness.

In cases where the submitted proposal has been accepted but lacks the completeness required by the project officer, finalized negotiations to remove the problem areas should be directly addressed in the work plan showing the details of the work to be done.

The work plan must be submitted to the project officer before any work is begun by the contractor. The plan can be accepted in draft form, which will allow for minor changes prior to the final plan's acceptance and approval.

2.4 Management Commitment to Quality Control

No quality control program, regardless of the amount of planning or level of effort expended, will be effective without the explicitly visible support of top management. The support should be expressed initially as the project gets underway and periodically throughout the duration of the program. The support of top management then filters down through middle and lower management to the operators, resulting in a program where QC is practiced on a day-to-day basis, rather than being an additional program or nuisance. Quality control must be built-in, functional area within the total program, and this is not possible without continuing obvious management support.

2.5 Quality Control in the Organization Structure

Support for quality control is most visible when the organizational structure has provision for personnel whose authority and responsibilities lie in the area; i.e., a quality control coordinator (QCC) and/or any other staff appropriate to the program. The QCC is responsible for the organization's entire QC program, and this person's judgment determines the effectiveness of the program. The basic function of the QCC should be the fulfillment of the QC objective of management in the most efficient and economical manner

commensurate with insuring continuing completeness, accuracy, and precision of the data produced. The responsibilities and authority of the QCC are detailed in the Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. 1, Principles, EPA-600/9-76-005.*

The QCC should have, within the main organizational structure, a subordinate organization for QC activities (auditing, calibration, quality control). He should have authority for assignment of QC duties and for coordination of the entire program, and must not be directly subordinate to operational personnel in the project.

2.6 Assessment of Quality Control Requirements

The establishment of a QC program for a demonstration project requires first of all the setting, in as quantitative a manner as possible, of project objectives. The desired precision and accuracy of each measurement should be specified, as well as the technical means of attaining this degree of data quality; i.e., the tasks to be performed. Once this is done, it is efficient to group the tasks organizationally and assign responsibility for the QC function to each task group. It is inevitable that problems are incurred in each step of the planning and establishment of the QC program. Some of these cannot be resolved until the program enters the functional stage. What is important initially is that these problems be identified and clearly stated, so that they can be resolved as quickly as possible once the program gets underway.

2.7 Specific Areas of Concern for Demonstration Project Quality Control Programs

A quality control program for a demonstration project serves to:

1. Evaluate the overall adequacy of the project insofar as data quality is concerned;
2. Identify potential as well as existing problems in the data-producing system, from measurement to data reduction;

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*Some of the general discussion of QC programs in this report has been taken from this document.

3. Stimulate research into and discussion of alternative methods for obtaining data of the required quality.

It is advisable to delineate a number of important aspects of the project which have direct bearing on data quality, and to discuss each of these in some detail.

2.7.1. Facilities and Equipment

An obvious beginning point in the assessment of an ongoing program is a general survey of the facilities and equipment available for day-to-day operation of the project. Are they adequate for the job at hand? Do standards exist for evaluation of facilities, equipment, and materials?

The laboratories, data processing sections, and other operation areas should be neat and orderly, within common-sense limits imposed by the nature of the facility. A neat, well-organized laboratory area serves to inspire neatness and organization among the laboratory workers.

Good laboratory maintenance, particularly for certain types of instrumentation, requires complete manuals, kept in a convenient place so that they are readily available to appropriate personnel. Responsibility for keeping up with all necessary manuals should be given to an individual, with the understanding that he must devise a system (checkin-checkout) for quick location of each document.

2.7.2 Configuration Control

The documentation of design changes in the system must be carried out unfailingly. Procedures for such documentation should be written, and be accessible to any individual responsible for configuration control.

2.7.3 Personnel Training

It is highly desirable that there be a programmed training system for new employees. This system should include motivation toward producing data of acceptable quality standards. This is to be preferred to on-the-job training, which may be excellent or slipshod, depending upon a number of circumstances.

A thorough personnel training program should focus particular attention on those people whose work directly affects data quality (calibration personnel, bench chemists, etc.). These people must be cognizant of the quality standards fixed for the project and the reasons for those standards. They must be made aware of the various ways of achieving and maintaining quality data. As these people progress to higher degrees of proficiency, their accomplishments should be reviewed and then documented.

2.7.4 Documentation Control

Procedures for making revisions to technical documents must be clearly written out, with the lines of authority indicated. The revisions themselves should be written and distributed to all affected parties, thus insuring that the change will be implemented and will become permanent.

2.7.5 Control Charts

Control charts are essential as a routine day-to-day check on the consistency or "sameness" of the data precision. A control chart should be kept for each measurement that directly affects the quality of the data. Typically, control charts are maintained for duplicate analyses, percent isokinetic sampling rate, calibration constants, and the like. An example control chart is given as figure 1. The symbol σ (sigma) represents a difference, d, of one standard deviation unit in two duplicate measurements, one of which is taken as a standard, or audit value. Two σ is taken as a warning limit and 3σ as a control limit.

2.7.6 In-Process Quality Control

During routine operation, critical measurement methods should be checked for conformance to standard operating conditions (flow rates, reasonableness of data being produced, and the like). The capability of each method to produce data within specification limits should be ascertained by means of appropriate control charts. When a discrepancy appears in a measurement method, it should be analyzed and corrected as soon as possible.

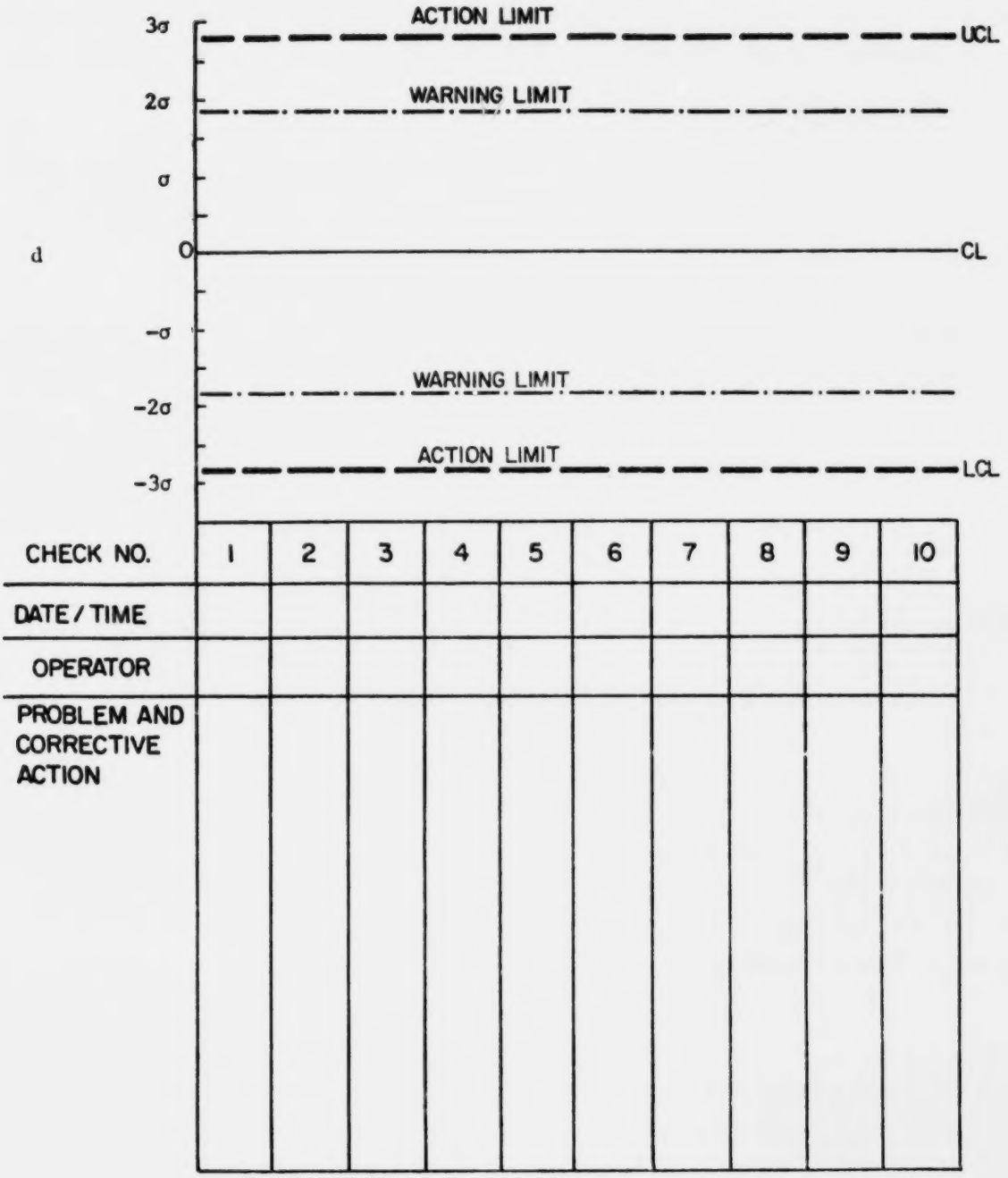

Figure 1. Standard quality control chart.

2.7.7 Procurement and Inventory Procedures

There should be well-defined and documented purchasing guidelines for all equipment and reagents having an effect on data quality. Performance specifications should be documented for all items of equipment having an effect on data quality. In the case of incoming equipment, there should be an established and documented inspection procedure to determine if procurements meet the quality assurance and acceptance requirements. The results of this inspection procedure should be documented.

Once an item has been received and accepted, it should be documented in a receiving record log giving a description of the material, the data of the receipt, results of the acceptance test, and the signature of the responsible individual. It is then placed in inventory, which should be maintained on a first-in, first-out basis.

2.7.8 Preventive Maintenance

It is most desirable that preventive maintenance procedures be clearly defined and written for each measurement system and its support equipment. When maintenance activity is necessary, it should be documented on standard forms maintained in log books. A history of the maintenance record of each system serves to throw light on the adequacy of its maintenance schedule and parts inventory.

2.7.9 Reliability

The reliability of each component of a measurement system relates directly to the probability of obtaining valid data from that system. It follows that procedures for reliability data collection, processing, and reporting should be clearly defined and in written form for each system component. Reliability data should be recorded on standard forms and kept in a log book. If this procedure is followed, the data can be utilized in revising maintenance and/or replacement schedules.

2.7.10 Data Validation

Data validation procedures, defined ideally as a set of computerized and manual checks applied at various appropriate levels of the measurement process,

should be clearly defined, in written form, for all measurement systems. Criteria for data validation must be documented. The required data validation activities (flow-rate checks, analytical precision, etc.) must be recorded on standard form in a log book.

Any demonstration project should, on a random but regular basis, have quality audits performed by in-house personnel. These audits must be independent of normal project operations, preferably performed by the QCC or appointees of the QCC. The audits should be both qualitative and quantitative (i.e., they should include both system reviews and independent measurement checks). For the system review, a checklist is desirable to serve as a guide for the reviewer. Such a checklist is included as appendix A of this report.

The quantitative aspect of the audit will vary depending on the nature of the project. Some guidelines for quantitative audits are given in appendix B.

2.7.11 Feedback and Corrective Action

Closely tied to the detection of invalid data is the problem of establishment of a closed loop mechanism for problem detection, reporting, and correction. Here it is important that the problems are reported to those personnel who can take appropriate action. A feedback and corrective action mechanism should be written out, with individuals assigned specific areas of responsibility.

2.7.12 Calibration Procedures

Calibration procedures are the crux of any attempt to produce quality data from a measurement system. For this reason it is extremely important that the procedures be technically sound and consistent with whatever data quality requirements exist for that system. Calibration standards must be specified for all systems and measurement devices, with written procedures for assuring, on a continuing basis, traceability to primary standards. Since calibration personnel change from time to time, the procedures must be, in each instance, clearly written in step-by-step fashion. Frequency of calibration should be set and documented, subject to rescheduling as the data are reviewed. Full documentation of each calibration and a complete history of calibrations performed on each system are absolutely essential. This permits a systematic review of each system reliability.

3.0 GUIDELINES FOR DEMONSTRATION PROJECT QUALITY ASSURANCE PROGRAMS

3.1 General Statements

The objective of quality assurance is to independently assess the quality control program of the project. This assessment should normally take two major forms: (1) a qualitative audit (systems review), and (2) a quantitative performance audit. These are discussed in detail below, as sections 3.2 and 3.3 respectively.

The frequency of a qualitative and/or a performance audit obviously should be dictated by the specific project. It is recommended that a minimum frequency be once each calendar year. The initial systems review and performance audit should take place within the first quarter of the first project year. Subsequent scheduling should be dependent on the requirements of management and the apparent quality of the day-to-day data being obtained. More frequent auditing may be necessary in the initial stages of the project.

3.2 The Qualitative Audit

The objective of the qualitative audit is to assess and document facilities; equipment; systems; recordkeeping; data validation; operation, maintenance, and calibration procedures; and reporting aspects of the total quality control program for demonstration projects. The review should accomplish the following:

1. Identify existing system documentation--i.e., maintenance manuals, organizational structure, operating procedures, etc;
2. Evaluate the adequacy of the procedures as documented;
3. Evaluate the degree of use of and adherence to the documented procedures in day-to-day operations based on observed conditions (auditor) and a review of applicable records on file.

To aid the auditor in performing the review, a checklist is included as appendix A. This checklist will allow for systematic appraisal of the areas mentioned above.

3.3 The Performance Audit

In addition to a thorough on-site qualitative audit, quantitative performance audits should be periodically undertaken at each demonstration project. The objective of these audits is to evaluate the quality of project data by independent measurement techniques. It is convenient to classify the major measurement methods into three areas: physical measurements, gas stream measurements, and liquid stream measures (the latter including analysis of any suspended solids). Appendix B lists in matrix form a number of standard techniques for auditing in the three major areas just mentioned. Table 1 of appendix B is a compilation of commonly measured physical properties, with a selection of possible measurement, calibration, and audit techniques. Table 2, concentrating on analysis of gas effluent streams, lists the material to be analysed and measurement, calibration, and audit techniques for that material. Finally, table 3 very briefly and generally deals with measurement methods appropriate to liquids and solids. The specific techniques vary widely from project to project, but the audit technique generally involves use of control (reference) samples of known composition and/or splitting a sample among several laboratories for independent analyses.

3.4 Material Balances

Material balances serve as a gross indication of the quality of the total measurement system complex of the project. The extent of closure will be directly related to the precision and bias of each measurement taken. In general, both physical measurements of flow rates, temperatures, pressured (and so on), and chemical analysis of material composition will bear on the degree of closure attained. The achievable extent of closure must be estimated for each project and used as a target figure. The frequency with which material balances are run is related to how successful one is in attaining the estimated closure over a significant period of time.

3.5 Assessment of Data Quality

Standard methods exist for estimation of the precision and accuracy of measurement data. Efficient usage of the audit data requires that a rationale

be followed which gives the best possible estimates of precision and accuracy within the limits imposed by timing, sample size, etc.

For a given measurement, the difference between the field (or plant) and the audited results,

$$d_j = X_j - X_{aj},$$

is used to calculate a mean and standard deviation as follows:

$$\bar{d} = \sum_{j=1}^{n} d_j/n \ ,$$

$$s_d = \sum_{j=1}^{n} (d_j - \bar{d})^2/(n - 1)^{1/2}$$

where $\bar{d}$ is an estimate of the bias in the measurements (i.e., relative to the audited value). Assuming the audited data to be unbiased, the existence of a bias in the field data can be checked by the appropriate t-test, i.e.,

$$t = \frac{\bar{d} - 0}{s_d/n} \ .$$

If t is significantly large, say greater than the tabulated value of t with n - 1 degrees of freedom, which is exceeded by change only 5 percent of the time, then the bias is considered to be real and some check should be made for a possible cause of the bias. If t is not significantly large, then the bias should be considered zero, and the accuracy of the data is acceptable.

The standard deviation, s_d, is a function of both the standard deviation of the field measurements and of the audit measurements. Assuming the audit values to be much more accurate than the field measurements, then s_d is an estimate of $\sigma\{X\}$, by using the statistical test procedure

$$\frac{X^2}{f} = \frac{s_d^2}{s^2_{\{X\}}}$$

where X^2/f is the value of a random variable having the chi-square distribution with $f = n - 1$ degrees of freedom. If X^2/f is larger than the tabulated value exceeded only 5 percent of the time, then it would be concluded that the test procedure is yielding more variable results due to faulty equipment or operational procedure.

The measured values should be reported along with the estimated biases, standard deviations, the number of audits, n, and the total number of field tests, N, sampled ($n \leq N$). Estimates (such as s_d and $\bar{d}$) which are significantly different from the assumed population parameters should be identified on the data sheet.

The t-test and X^2-test described above are used to check on the biases and standard deviations separately.

Other statistical techniques exist which may apply to specific projects (or to highly specialized areas of a given project). It is usually worthwhile to acquire the services of a statistical consultant in order to more effectively treat the available data.

3.6 Assessment and Modification of the Ongoing Data Quality Program

The guidelines put forth in the preceding sections serve as a basis for development of a data quality program specific to the needs of a particular project. A program should not be attempted without a thorough study of the entire facility, supplemented by at least one site visit. It is to be desired that provision for QC be made from the project's inception. The EPA project officer's responsibility is then to see that a program of adequate QC practices is incorporated into the day-to-day project operations, along with periodic QA audits conducted by outside organizations.

Implementation of the program, at whatever point in the lifetime of the project, exposes weaknesses of approach and problems that were not anticipated in the planning stages. Certainly it is necessary to maintain maximum flexibility of approach as the interface with project realities is made. The

Shawnee report documents several problems of implementation and suggests procedures for avoiding those problems in future efforts. They are also discussed in the final section of this report.

Generally, one should expect that a degree of modification would be required in the areas listed below:

1. audit instrumentation and general equipment requirements,
2. sampling frequencies, and
3. audit personnel requirements.

Experience must always be the final judge of the effectiveness of a data quality program, as one monitors data quality on a continuing basis.

4.0 A SHORT-TERM QUALITY ASSURANCE PROGRAM IMPLEMENTED AT THE SHAWNEE SCRUBBER FACILITY

A quality assurance program was implemented at the wet scrubber facility, Shawnee steam Plant, Paducah, Kentucky. This program was carried out by personnel of the Research Triangle Institute, Research Triangle Park, North Carolina. The program consisted of two site visits of approximately 1 week each, occurring October 28-31 and November 17-21, 1975. The first visit was largely occupied with the qualitative audit, using as a guide the checklist provided as appendix A. An evaluation was made of equipment needs for the second visit, when the quantitative performance audit was conducted. This section presents the results of both the qualitative review and the performance audit, with recommendations for a QA program at the facility.

4.1 The Control Laboratory

4.1.1 Measurement of pH

One of the major elements for control of the scrubber is pH measurement. For this reason a significant part of the RTI effort was spent in observing and verifying the TVA techniques for pH determination at scrubber inlet and outlet.

A portable pH system is used several times a day by TVA operators to check the control room readings obtained from inline pH sensors. These devices are permanently situated in pots through which slurry continuously circulates when the scrubber is operating. Four such sensors monitor the inlet and outlet pH for the TCA and Venturi scrubbers.

A series of direct comparison pH measurements were made on November 19-20, 1975. RTI and TVA personnel made simultaneous measurements of slurry pH. The operators were instructed to carry out their measurements routinely, from standardization to cleanup. Also during this period the inline probes were removed from their pots and immersed in pH 5 and 6 buffers. Both RTI and TVA long-lead probes were put into these same buffers, after independent standardization. The values obtained in the buffers and in slurry are summarized in table 1. Measurements to the nearest 0.01 pH unit were made using the RTI Acumet pH meter. Readings of such precision were not possible with the TVA

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Table 1. Comparisons* of pH

TEST POINT	pH RTI (portable)	pH TVA (portable)	pH TVA (inline)	Temperature (°Centrigrade)	Date (mo/day/yr)	Time (hours)
1816[1]	5.25	5.3	5.16	54	11/19/75	11:15
	5.13	5.2	5.29	54	11/19/75	15:30
	5.05[5**]	4.9	5.04	16	11/20/75	08:45
	5.24	5.2	5.34	54	11/20/75	09:10
	5.28	5.2	5.38	50	11/20/75	09:45
	5.34	5.3	5.34	51	11/20/75	11:15
	5.06	5.1	5.25	50	11/20/75	15:15
1825[2]	4.92	4.9	5.17	53	11/19/75	11:30
	4.83	4.9	5.02	54	11/19/75	15:30
	4.77	4.8	5.08	50	11/20/75	15:15
2816[3]	5.03[5]	—	4.82	21	11/19/75	10:00
	6.02[6]	6.0	5.77	21	11/19/75	10:30
2825[4]	5.03[5]	5.0	4.99	21	11/19/75	10:50
	6.01[6]	6.0	6.12	21	11/19/75	11:00

1 Venturi effluent hold tank.
2 Venturi outlet.
3 TCA effluent hold tank.
4 TCA outlet.

*Unless otherwise noted, measurements are on slurry in inline probe pots.

**Superscript number indicates measurement of a buffer solution of pH 5 or 6.

Orion pH meter, where estimates of 0.1 pH unit were made. Actual scale markings on the TVA meter were at 0.2-pH unit intervals.

Two observations are in order, after study of table 1:

1. The RTI and TVA portable (long-lead) pH systems agreed within 0.1 unit or better in every comparison made. This verifies the accuracy of the TVA portable system, since the RTI system was standardized against certified buffer. Operator reading errors are probably the largest error source.

2. The inline system readings differed from RTI readings from 0 to 0.3 pH unit, with the mean difference being 0.14 pH unit over 14 readings. Certainly it would be unwise to dwell on the significance of the statistics of such a brief study. One point can be made, however, with respect to the confidence placed in the inline pH readings, as follows: it appears unlikely that, using the present system, pH measurements on the slurry can be made to better than 0.1 pH unit. There are a number of factors which militate against greater accuracy, the major one probably being the nature of the slurry itself. This viscous, highly abrasive suspension tends to clog lines, coat out on probe surfaces, etc., making reproducible measurements quite difficult. The non-equilibrium mixture of reactive chemicals has a pH that will change on removal from the scrubber proper; i.e., as it flows into the pots within which measurements are made.

Another factor is the difficulty of standardization of inline probes. The present system calls for probe removal, cleaning, and standardization roughly each 2 days. The accuracy of the pH reading is surely dependent on the condition of the probe surface, and restandardizing is ideally done shortly before each measurement.

4.1.2 Slurry Analysis

As a check on the reliability of the chemical analysis phase of the scrubber operation, a series of slurry samples was collected* and sent to several other laboratories for independent analysis of both the liquid phase and suspended solids.

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*All samples were taken from the venturi effluent hold tank.

The laboratories originally selected for participation in this phase of the audit were two TVA laboratories (Chattanooga and Muscle Shoals), E/ETB (EPA-RTP), and RTI. The E/ETB laboratory later declined to participate in the project.

Each laboratory was given five 1-ℓ samples of slurry, which were taken concurrently with control laboratory samples. After filtering and drying the solid, it was analyzed for calcium, magnesium, and total sulfur. The filtrate was analyzed for calcium, magnesium, sodium, potassium, and chloride.

Complete results of the analyses are given as appendix C. These results are given in matrix form, both by laboratory and by element. This report will present a limited statistical analysis of the data and will comment on the techniques used by each laboratory.

The five slurry samples were taken over a 36-hour period, all from the effluent hold tank. There was little apparent change in the slurry composition over this period of time. Taking a simple numerical average of the five analyses for each element yields a number which itself has little significance, since it represents the combined effect of analytical uncertainty and slurry composition change over 36 hours. The rationale for obtaining such an average is that, if the analytical technique exhibits a bias, this will result in a number that is correspondingly biased. Thus a comparison can be made among the participating laboratories and the various analytical techniques. Table 2 is a matrix of these averages, with the analytical technique used by the laboratory given underneath the number. A few observations are in order:

1. Analysis results for calcium in the solid were extremely close among the Shawnee, Chattanooga, and Muscle Shoals laboratories, but RTI obtained a considerably lower value by AA. The Shawnee XRF standard value for calcium was established by sending portions of the standard to various TVA laboratories, including the Chattanooga and Muscle Shoals facilities. Also, not that these two laboratories both used the same technique, EDTA titration. Further work would be required to determine which technique, EDTA or AA, is inherently more accurate.
2. Results for magnesium show the expected large variation for an element present in low concentration.

Table 2. Mean values for slurry analyses, by laboratory

LABORATORY / ELEMENT	SHAWNEE	CHATTANOOGA	MUSCLE SHOALS	RTI
Ca (CaO) wt % in solid	23.19 XRF	22.93 EDTA	23.16 EDTA	19.72 AA
Mg (MgO) wt % in solid	0.29 XRF	0.60 EDTA	0.25 AA	0.42 AA
Total Sulfur (SO_3) wt % in solid	31.99 XRF	30.00 $BaSO_4$ ppt.	30.74 $BaSO_4$ ppt.	33.01 $BaSO_4$ ppt.
Ca, ppm in liquid	1929 AA	1700 EDTA	1759 EDTA	1800 AA
Mg, ppm in liquid	697 AA	772 EDTA	736 AA	932 AA
Na, ppm in liquid	71 AA	65 FE	39 FE	74 AA
K, ppm in liquid	128 AA	108 FE	56* FE	106 AA
Cl, ppm in liquid	3580 Pot. Titration	3601 Volhard	3680 $AgNO_3$ titration	3780 Volhard

XRF = X-ray fluorescence

EDTA = Ethylenediaminetetracetic acid titration

$BaSO_4$ ppt. = Precipitation as barium sulfate

AA = Atomic absorbance

FE = Flame emission

* Value discarded, second RTI value (different RTI laboratory) of 111 ppm used for statistical purposes.

3. Total sulfur determinations were consistent between Chattanooga and Muscle Shoals; RTI's value was high and Shawnee's came in between, with no large discrepancies.
4. Results in the liquid phase were relatively consistent, although the Muscle Shoals laboratory obtained extremely low numbers for sodium and potassium. Chattanooga, which also used flame emission, got results consistent with the AA determinations of Shawnee and RTI.
5. Chloride determinations showed good consistency across all laboratories, although an interesting sidelight is that some preliminary determinations at RTI, using a chloride-sensing electrode, gave results that were high by roughly 100 percent. This result was duplicated by a second, non-RTI laboratory, indicating that the electrical environment of the slurry liquid phase was unsuitable for chloride determination by ion-selective electrode.

Table 3 singles out the Shawnee control laboratory results for comparison with the mean results from the other three laboratories. Close agreement exists for the most critical elements--calcium (both liquid and solid), total sulfur (solid), and chloride (liquid). Magnesium in the solid showed the greatest variation among the comparison laboratories (43 percent) and between Shawnee and the mean of the comparison laboratories (31 percent). Across the board, these results indicate the Shawnee control laboratory is performing routine analyses at about the $\pm$20 percent level, referenced to comparable laboratories. In particular, it appears to be obtaining rather good accuracy ($\pm$5 to 10 percent) in its calcium, sulfur, and chloride analyses. These results are consistent with material balance closures of 5 to 15 percent that have been repeatedly obtained by Bechtel Corporation.

It should be stressed that the percentages quoted above are simple estimates based upon direct comparisons between the Shawnee results and the mean of the cooperating laboratories' results. They are not confidence levels. It would be appropriate to carry out detailed statistical work only if considerably more data were obtained.

Table 3. Comparison of Shawnee results with mean value of cooperating laboratories

ELEMENT	SHAWNEE RESULTS	MEAN OF CHATTANOOGA, MUSCLE SHOALS & RTI RESULTS	STANDARD DEVIATION ABOUT MEAN	COEFFICIENT OF VARIATION ABOUT MEAN (%)	(SHAWNEE/MEAN) x 100 -100 (%)
Ca (CaO) wt % in solid	23.19	21.94	1.92	8.8	+ 5.7
Mg (MgO) wt % in solid	0.29	0.42	0.18	43	- 31
Total Sulfur (SO_3) wt % in solid	31.99	31.25	1.57	5.0	+ 2.4
Ca, ppm in liquid	1929	1753	50	2.9	+ 10.0
Mg, ppm in liquid	697	813	104	12.8	- 14.3
Na, ppm in liquid	71	59	18.2	31	+ 19.7
K, ppm in liquid	123	108*	2.5	2.3	+ 18.5
Cl, ppm in liquid	3580	3687	90	2.4	- 2.9

* Low Muscle Shoals results thrown out, second RTI result (from another RTI laboratory) of 111 ppm used in averaging.

A final point is that the Shawnee ionic imbalances for solid and liquid were typically 2 to 3 percent and 10 to 15 percent respectively, both biased negatively during the sampling period. The close solid ionic balance indicates either accurate analyses or balancing positive and negative ion analytical errors. Results of this study appear to validate the accuracy of the analytical procedures used on the slurry solid.

The negative ionic imbalance in the liquid phase analyses cannot be rationalized by the data obtained from this comparison study.

4.1.3 Overall Laboratory Evaluation

The control laboratory operation appears to be adequate for the routine analytical work it performs. It has no formal quality control program, but bad data may be flagged by either TVA or Bechtel personnel. Acceptance limits on data are not formalized, but "reasonableness" is the experience-based criterion.

There are problems associated with the lack of operator training programs, incentives for superior performance and the like, but so long as the laboratory operations remain strictly routine these problems are not likely to seriously hamper the program.

Equipment and instrumentation is appropriate for the type of work done, and it is maintained on a regular basis (largely by service contracts).

4.2 Gas Stream Sampling

4.2.1 Particulate Mass Loading

Side-by-side duplicate runs were not attempted. The entire sampling procedure was observed, with critical techniques checked repeatedly, during the site visits. Overall performance was evaluated using a checklist. On a scale of 1 to 5, ranging from unacceptable to excellent, the Shawnee particulate loading technique was rated 3 (acceptable). A major problem appeared to be the failure of Shawnee personnel to carry out adequate leak-checking of the sampling train. Specific comments are given below.

4.2.1.1 Pitot tube comparison.

A comparison of TVA and RTI pitot tubes was performed at the Venturi inlet only. A check of the outlet tube was not carried out due to the outlet tube misalignment ($\geq$ 30°) along its roll axis.

Side-by-side measurements were performed. Based upon comparison with the RTI (NBS calibrated) pitot tube, the TVA tube Cp factor was 0.879. The assumed value was 0.850. The difference was considered to be negligible.

4.2.1.2 Temperature measurement.

A system capable of measuring the stack gas temperature to within 1.5 percent of the minimum absolute stack temperature is required. The temperature-measuring system (inlet sampler) was checked versus a calibrated thermocouple and was found to be within 1 percent.

4.2.1.3 Moisture measurement.

The impinger section of the EPA sampling train is intended to collect moisture from the sample gases for determination of moisture content. The last impinger contains silica gel to adsorb the water vapor not condensed in the first two impingers. The moisture content of the sample gas leaving the silica gel impinger increases as the exit gas temperature rises. Also, the exit gas moisture content will increase as the sample train vacuum increases at any one sample temperature. Moisture not collected by the condensation system is incorrectly measured as dry gas by the dry test meter and the error is carried through the isokinetic and grain loading calculations. However, if the exit gas temperature is held below 25° C and the rain vacuum is held below 380 mm of Hg, the resulting error in the sample volume will be less than 2 percent. A single RTI reading of exit gas temperature was 22° C.

There was evidence of significant moisture accumulation in the silica gel, indicating the presence of some water vapor in the total gas volume measured. This does not likely introduce a large error into the technique, although it would be advisable to make quantitative or semiquantitative checks on the actual water volume collected versus water content of the stack gas. This is not presently being done at Shawnee.

4.2.1.4 Volume measurement.

The sampling train was checked for accuracy of volumetric measurement with a calibrated dry test meter (1 cf/revolution) which had been previously calibrated versus a 1-cf wet test meter. The RTI meter was connected directly to the Shawnee probe tip, so that the actual volume intake at the probe was measured. RTI volume was 15.8 percent lower than TVA volume, indicating a rather large positive bias in the TVA measurement. Critical examination of the TVA sampling system led to the conclusion that the bias could be attributed to leaks in the system (broken or cracked polycarbonate impinger tubes, loose probe tip, etc.). Leakage rate was estimated to be 0.36 cfm at 380 mm of Hg vacuum.* Inaccuracies in volume measurements appear directly in the concentration and particulate mass emission rate determinations.

A probe tip diameter check was made with a micrometer. The range of the diameter measurements was 0.7 mm, indicating a severely out-of-round nozzle which should be repaired or replaced. The estimated nozzle area was calculated to be roughly 20 percent lower than the assumed area (0.583 cm^2 calculated, 0.7125 cm^2 assumed). An error in the nozzle diameter is quadrupled in the process of determining isokinetic sampling rates and is doubled in the percent of isokinetic sampling calculation. The percent isokinetic, as calculated with respect to the above errors in volume measurement and nozzle diameter could result in either a positive or negative bias, depending upon which factor predominates.

4.2.2 Sulfur Dioxide Concentration Determinations

Sulfur dioxide concentrations at the wet limestone scrubber facility are determined by means of du Pont Model 400 photometric analyzers. The analyzers continuously monitor inlet and outlet gas streams of the venturi and TCA units.

The RTI audit team collected a total of 23 gas samples, all collected at the venturi inlet. Thirteen of these samples were analyzed by a modified barium chloranilate (colorimetric) method, the remaining 10 by sodium hydroxide

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*TVA leak-checking was not observed by the audit team. A thorough leak-check would surely have detected such a significant leak-rate.

titration. Results are given in table 4. The average bias of the photometric method with respect to the wet chemical methods was +6.9 percent, with a standard deviation of 8.7 percent. These results indicate that the du Pont analyzer at the venturi inlet is yielding data of high quality. At the 95 percent confidence level, an individual photometric determination should have a precision of ±18 percent of the mean concentration, biased 7 percent high on the average.* Due to the time limitation of the audit team, it was not possible to run checks on the other three analyzers.

4.3 Process Instrumentation

A series of calibration checks on the electronic instrumentation was scheduled. Some checks and observations were not carried out because of TVA personnel work schedules, but enough was accomplished for a judgment to be made as to the quality of the instrumentation facilities.

Three types of sensors (temperature, differential pressure, and flow rate) are the primary sources of measurement information being recorded and used for the scrubber's mechanical operation control, and four readout devices are employed for visual display of the output signals. The methods of test and calibration are simple, using rudimentary sources of stimuli for sensor examination. Straightforward electrical current measuring instruments are used to monitor currents produced by the transmitters. As performed, the tests are sufficient to maintain the quality of measurement to the degree established by the manufacturers in their design specifications.

The performance of equipment over time can best be judged by a review of accurate records which clearly show a life history of each item having a functional part in the operation of a system. The Shawnee facility was judged deficient in recordkeeping for its instrumentation. In spite of this, it was felt that the electronic devices used for physical measurements were being maintained sufficiently to provide pressure, level, and flow information to a ±2 percent tolerance of desired nominal values, and temperature information to a ±10 percent tolerance of desired information (temperature sensors can be

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*This assumes no bias in the wet methods.

Table 4. Comparison of SO_x determinations

Date	Sample Number	Sample Time	Sampling Train No.1: SO_2 by Barium-Chloranilate (ppm)	Sampling Train No.1: SO_x Contained in Isopropranol Scrubber (ppm)	Sampling Train No.1: Total	Sampling Train No.2: SO_x by NaOH Titration*	Sampling Train No.2: SO_2 by TVA (DuPont Analyzer)	$\frac{TVA-RTI}{RTI} \times 100$
11/18/75	1	17:15-17:25				2562	2750	+7.3
11/18/75	2	17:35-17:45				2252	2750	+9.0
11/19/75	3	10:15-10:23	4187	543	4730		3625	- 23.3
11/19/75	4	10:42-10:50	2736	650	3386		3585	+5.9
11/19/75	5	11:07-11:21	Sample Voided	---	----		3366	---
11/19/75	6	12:08-12:17	Sample Voided	---	----		3167	---
11/19/75	7	12:43-12:49				3053	3051	-0.1
11/19/75	8	12:53-13:02	2261	530	2791		3046	+9.1
11/19/75	9	13:39-13:47				2832	3016	+6.5
11/19/75	10	13:50-13:59	2172	563	2735		2985	+9.1
11/19/75	11	14:22-14:31				2812	2998	+6.6
11/19/75	12	14:32-14:41	2309	530	2839		2947	+3.8
11/19/75	13	15:06-15:15				2757	2893	+4.9
11/19/75	14	15:17-15:27	1958	551	2509		2909	+ 15.9
11/20/75	15	09:16-09:27	1582	523	2105		2506	+ 19.0
11/20/75	16	09:30-09:40				2435	2509	+3.0
11/20/75	17	09:53-10:04	1827	643	2470		2505	+1.4
11/20/75	18	10:05-10:15				2385	2508	+5.2
11/20/75	19	10:22-10:33	1750	610	2360		2570	+8.9
11/20/75	20	10:41-10:52				2459	2600	+5.7
11/20/75	21	10:58-11:09	1724	541	2265		2606	+ 15.1
11/20/75	22	11:11-11:22				2294	2648	+ 15.4
11/20/75	23	11:37-11:48	1690	637	2327		2719	+ 16.8

* SO_2 analyzer accepted on basis of a total acid determination (TVA analysis)

Average Bias: +6.9%

Std. Dev.: 8.69%

95% Confidence Interval ± 18.08%

calibrated to a ±2 percent tolerance of a known temperature--the inaccuracies are estimated to be high because of the lack of knowledge of the thermodynamics of the stack gases being measured).

4.4 Recommendations

Specification of a quality control program for the Shawnee scrubber project does not fall within the scope of this report. The recommendations made in the following paragraphs apply to the implementation of a (qualitative and performance audit) QA program.* This type of program normally should be carried out by an organization which has no special interest in the data; i.e., no self-interest to protect and no preconceptions as to the quality of the information forthcoming. On the other hand, the organization should be reputable and well qualified to carry out the type of auditing program desired.

In the case of EPA demonstration projects, EPA may wish to contract a third party to handle the audit program, or it may handle the program by means of its own QA staff. In either case, it is quite important that the auditing be done competently and objectively.

It is recommended that the wet limestone scrubber operation located at the Shawnee steam-electric plant be externally audited twice each calendar year. Timing of the audit program, which normally should take 1 work week, should be coordinated among the auditing team, EPA, TVA, and the Bechtel Corporation. Some advance notice is necessary in order to insure cooperation of operational personnel. It is not recommended that the audits be scheduled on a regular basis, since by definition an audit is conducted without extensive "preparation" at the project being audited. Advance notice to EPA and Bechtel supervisory staff should be at least 2 weeks, so that the audit team can be apprised of special test and analysis schedules which may alter its audit procedure or cause postponement of the audit itself. Advance notice to TVA staff (senior chemist, instrumentation foreman) should be at least 1 week.

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*It is important that the Shawnee project continue to develop its own QC program internally.

It is recommended that the audit team concentrate its efforts in the following major areas:

1. Verification of pH measurements at inlet and outlet, on both TCA and venturi scrubbers. An accurate pH meter brought in by the audit team, with appropriate buffer solutions, should be used. Measurement of pH is critical to efficient process control at this facility.
2. Independent chemical analysis of slurry samples by several laboratories. A continuing audit program can aid in establishing acceptance limits and method biases.
3. Verification of particulate mass loading and sulfur dioxide measurement systems. If possible, side-by-side operation of TVA and audit team sampling trains should be carried out, with independent analyses of the collected samples. A wet chemical technique such as total acid titration should be used to check the SO_2 analyzer response. If a duplicate sampling train cannot be used by the audit team, then critical measurement parameters should be identified and checked. For stack sampling procedure, this includes (at a minimum):
   a. Sample volume measurement check by means of a calibrated wet test or dry gas meter;*
   b. Pitot tube (C_P factor) check by means of an NBS calibrated pitot tube;
   c. Thermometer and thermocouple checks with a calibrated temperature measurement system;
   d. Stack gas moisture content check by means of an absorbing impinger train.
4. Electronic checks on process instrumentation and physical measurement techniques.

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*For suggestions as to techniques available, see "Process Stream Volumetric Flow Measurement and Gas Sample Extraction Methodology," by Brooks and Williams. This manual (TRW Document No. 24916-6028-RU-00) was prepared under EPA Contract No. 68-02-1412, for the Process Measurements Branch of IERL.

5.0 EVALUATION OF THE SHORT-TERM QUALITY ASSURANCE PROGRAM AT SHAWNEE

The objective of this project was to devise, implement, and modify a general quality assurance program for IERL demonstration projects. It is appropriate then to evaluate the procedures which RTI used in its qualitative and performance audit at the Shawnee scrubber facility.

5.1 Qualitative Audit

The audit checklist (appendix A) is a valuable device for assuring a balanced review of each major area. For the control systems laboratory operations, the checklist was quite useful. It is recommended that either this checklist or a similar one be used each time a review is done. One note of caution is in order. No checklist can substitute for experience and common sense. Each facility will require a somewhat different approach by a QA team. The checklist alone will not suffice. It will be particularly appropriate with certain phases of a project, but in other areas a good part of the questionnaire will not be applicable.

5.2 Quantitative Performance Audit

The quantitive performance audit at Shawnee emphasized several potential problem areas of which an audit team should be cognizant. These are discussed in the subsections below.

5.2.1 Scheduling

If samples are to be taken for round robin purposes, it is important that the laboratory being audited perform complete analyses on each sample to be distributed to outside laboratories. This requires close supervision by the member of the auditing team responsible for sample collection. At Shawnee, the sampling schedule (at the time of the audit) called for analysis of the solid at certain times of the day, and both solid and liquid at other times. Because of the preoccupation of the senior chemist with routine operations, this schedule was not made clear and several samples (which required complete analyses for comparison purposes) were collected at times calling for solid

analysis only. When this was discovered, it was necessary to ship some surplus samples (which RTI was fortunate to have kept) back to the Shawnee laboratory for liquid analysis. Had the surplus samples not been available, it would have been necessary to collect a second series of samples at another time. This example serves to point out the need for complete understanding, on the part of both senior laboratory personnel and audit team personnel, of the requirements of the audit.

A related scheduling problem occurred when the Shawnee instrumentation foreman was unable to provide RTI with the assistance needed to complete calibration and testing of the facilities' electronic process monitoring equipment. It is of overriding importance that efficient scheduling of audit activities occur, since the total time allowed is normally a few days to 1 week. Unscheduled operational problems will occur which could not have been anticipated, and these must be handled as expeditiously as possible.

5.2.2 Equipment and Instrumentation

It should be unnecessary to emphasize the desirability of having reliable, high-quality audit equipment and instrumentation. Where feasible, the audit team should have duplicate (or at least equivalent) backup items of that equipment which is crucial to the audit. For example, if pH measurements are to be made, two probes should be on hand. If possible, two meters should also be available. During the Shawnee audit, a measurement problem developed due to a shielded probe lead making electrical contact with the meter chassis. This problem had not been discovered earlier because the meter had previously been used only in nongrounded environments (such as glass beakers). The metal slurry pots were directly grounded to the scrubber framework and a relatively large extraneous signal was being read by the meter. This signal, by its magnitude, swamped the circuit and pegged the meter dial each time a pH reading was attempted. Fortunately, the problem was diagnosed as a missing insulating washer, and a washer was fabricated in time to take a series of readings. It would have been much more desirable to have had a second meter immediately available, since pH comparisons were certainly an important aspect of this work.

Spare glassware (and other fragile items) is obviously desirable, since in an unfamiliar environment laboratory workers are more prone to make mistakes resulting in breakage.

5.2.3 Personnel Selection

An audit team must be competent and versatile. The RTI team assigned to the Shawnee project consisted of a physical chemist, an analytical chemist, and an electrical engineer. Each man knew his responsibility. An audit visit, because of its short duration, does not allow for inefficient use of personnel time or audit equipment. The services of the electrical engineer were useful when the pH meter malfunction was discovered, pointing up the advantages of a diversity of technical talent. A team composed entirely of chemists might not have been able to repair the meter in time to make the required number of measurements.

APPENDIX A

QUALITATIVE AUDIT CHECKLIST FOR DEMONSTRATION PROJECTS

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APPENDIX A QUALITATIVE AUDIT CHECKLIST FOR DEMONSTRATION PROJECTS

This checklist is designed to:

1. Identify existing system documentation; i.e., maintenance manuals, organizational structure, operating procedures, etc.
2. Evaluate the adequacy of the procedures as documented.
3. Evaluate the degree of use of and adherence to the documented procedures in day-to-day operations based on observed conditions (auditor) and a review of applicable records on file.

The checklist gives three descriptions to each facet of a quality control system. In all cases the "5" choice is the most desirable and effective mode of operation; "3" is marginal and tolerable; "1" is definitely unacceptable and ineffective as a mode of operation.

It is not always possible to describe accurately all options with only three choices. Therefore, a "2" or "4" rating may be selected if the evaluator feels that an in-between score is more descriptive of the actual situation.

After all the applicable questions are answered, an average is computed to give an overall indication of the quality system effectiveness.

Generally, a rating of 3.8 or better is considered acceptable.

A rating between 2.5 and 3.8 indicates a need for improvement but no imminent threat to project performance as it stands.

For the control laboratory, the results are as follows:

1. Of 82 check questions, 65 were answered on site;
2. Average score was 3.0 (5.0 maximum), indicating a satisfactory but not outstanding program as presently operated;
3. The control laboratory was judged weak in its quality control organization, procurement, and inventory procedures, and in its personnel training policy;
4. Strong points were its day-to-day "in-process" quality assurance, its calibration procedures, and its facilities and equipment.

The completed questionnaire, with indicated judgments in specific areas, is given herewith. These judgments are for the control laboratory operation only.

A.1 QUALITY ORGANIZATION

SCORE

(1.1) Overall responsibility for quality assurance (or quality control) for the organization is:

(a) Assigned to one individual by title (e.g., Quality Control Coordinator). 5

(b) Assigned to a specific group within the organization. 3

(c) Not specifically assigned but left to the discretion of the various operational, analytical, inspection, and testing personnel. 1

(1.2) The Quality Control Coordinator is located in the organization such that:

(a) He has direct access to the top management level for the total operation, independent of others involved in operational activities. 5

(b) He performs as a peer with others involved in operational activities, with access to top management through the normal chain of command. 3

(c) His primary responsibility is in operational activities, with quality assurance as an extra or part-time effort. 1

(1.3) Data reports on quality are distributed by the Quality Control Coordinator to:

(a) All levels of management.* 5

(b) One level of management only. 3

(c) The quality control group only. 1

(1.4) Data Quality Reports contain:

(a) Information on operational trends, required actions, and danger spots. 5

(b) Information on suspected data/analyses and their causes. 3

(c) Percent of valid data per month. 1

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*Management at appropriate levels in all applicable organizations such as subcontractors, prime contractor, EPA.

A.2 THE QUALITY SYSTEM

SCORE

(2.1) The quality control system is:

(a) Formalized and documented by a set of procedures which clearly describe the activities necessary and sufficient to achieve desired quality objectives, from procurement through to reporting data to the EPA/RTP. 5

(b) Contained in methods procedures or is implicit in those procedures. Experience with the materials, product, and equipment is needed for continuity of control. 3

(c) Undefined in any procedures and is left to the current managers or supervisors to determine as the situation dictates. 1

(2.2) Support for quality goals and results is indicated by:

(a) A clear statement of quality objectives by the top executive, with continuing visible evidence of its sincerity, to all levels of the organization. 5

(b) Periodic meetings among operations personnel and the individual(s) responsible for quality assurance, on quality objectives and progress toward their achievement. 3

(c) A "one-shot" statement of the desire for product quality by the top executive, after which the quality assurance staff is on its own. 1

(2.3) Accountability for quality is:

(a) Clearly defined for all sections and operators/analysts where their actions have an impact on quality. 5

(b) Vested with the Quality Control Coordinator who must use whatever means possible to achieve quality goals. 3

(c) Not defined. 1

A.2 THE QUALITY SYSTEM (continued)

SCORE

(2.4) The acceptance criteria for the level of quality of the demonstration projects routine performance are:

(a) Clearly defined in writing for all characteristics. 5

(b) Defined in writing for some characteristics and some are dependent on experience, memory and/or verbal communication. 3

(c) Only defined by experience and verbal communication. 1

(2.5) Acceptance criteria for the level of quality of the project's routine performance are determined by:

(a) Monitoring the performance in a structured program of inter- and intralaboratory evaluations. 5

(b) Scientific determination of what is technically feasible. 3

(c) Laboratory determination of what can be done using currently available equipment, techniques, and manpower. 1

(2.6) Decisions on acceptability of questionable results are made by:

(a) A review group consisting of the chief chemist or engineer, quality control, and others who can render expert judgment. 5

(b) An informal assessment by quality control. 3

(c) The operator/chemist. 1

A.2 THE QUALITY SYSTEM (continued)

SCORE

(2.7) The quality control coordinator has the authority to:

(a) Affect the quality of analytical results by inserting controls to assure that the methods meet the requirements for precision, accuracy, sensitivity, and specificity. 5

(b) Reject suspected results and stop any method that projects high levels of discrepancies. 3

(c) Submit suspected results to management for a decision on disposition. 1

A.3 IN-PROCESS QUALITY ASSURANCE

(3.1) Measurement methods are checked:

(a) During operation for conformance to operating conditions and to specifications, e.g., flow rates, reasonableness of data, etc. 5

(b) During calibration to determine acceptability of the results. 3

(c) Only when malfunctions are reported. 1

(3.2) The capability of the method to produce within specification limit is:

(a) Known through method capability analysis ($\bar{X}$-R Charts) to be able to produce consistently acceptable results. 5

(b) Assumed to be able to produce a reasonably acceptable result. 3

(c) Unknown. 1

(3.3) Method determination discrepancies are:

(a) Analyzed immediately to seek out the causes and apply corrective action. 5

(b) Checked out when time permits. 3

(c) Not detectable with present controls and procedures. 1

A.3 IN-PROCESS QUALITY ASSURANCE (continued)

SCORE

(3.4) The operating conditions (e.g., flow rate, range, temperature, etc.) of the methods are:

(a) Clearly defined in writing in the method for each significant variable. 5

(b) Controlled by supervision based on general guidelines. 3

(c) Left up to the operator/analyst. 1

(3.5) Auxiliary measuring, gaging, and analytical instruments are:

(a) Maintained operative, accurate, and precise by regular checks and calibrations against stable standards which are traceable to the U.S. Bureau of Standards. 5

(b) Periodically checked against a zero point or other reference and examined for evidence of physical damage, wear or inadequate maintenance. 3

(c) Checked only when they stop working or when excessive defects are experienced which can be traced to inadequate instrumentation. 1

A.4 CONFIGURATION CONTROL

(4.1) Procedures for documenting, for the record, any design change in the system are:

(a) Written down and readily accessible to those individuals responsible for configuration control. 5

(b) Written down but not in detail. 3

(c) Not documented. 1

A.4 CONFIGURATION CONTROL (continued)

SCORE

(4.2) Engineering schematics are:

(a) Maintained current on the system and subsystem levels. 5

(b) Maintained current on certain subsystems only. 3

(c) Not maintained current. 1

(4.3) All computer programs are:

(a) Documented and flow charted. 5

(b) Flow charted. 3

(c) Summarized. 1

(4.4) Procedures for transmitting significant design changes in hardware and/or software to the EPA project officer are:

(a) Documented in detail sufficient for implementation. 5

(b) Documented too briefly for implementation. 3

(c) Not documented. 1

A.5 DOCUMENTATION CONTROL

(5.1) Procedures for making revisions to technical documents are:

(a) Clearly spelled out in written form with the line of authority indicated and available to all involved personnel. 5

(b) Recorded but not readily available to all personnel. 3

(c) Left to the discretion of present supervisors/managers. 1

A.5 DOCUMENTATION CONTROL (continued)

SCORE

(5.2) In revising technical documents, the revisions are:

(a) Clearly spelled out in written form and distributed to all parties affected, on a controlled basis which assures that the change will be implemented and permanent. 5

(b) Communicated through memoranda to key people who are responsible for effecting the change through whatever method they choose. 3

(c) Communicated verbally to operating personnel who then depend on experience to maintain continuity of the change. 1

(5.3) Changes to technical documents pertaining to operational activities are:

(a) Analyzed to make sure that any harmful side effects are known and controlled prior to revision effectivity. 5

(b) Installed on a trial or gradual basis, monitoring the product to see if the revision has a net beneficial effect. 3

(c) Installed immediately with action for correcting side effects taken if they show up in the final results. 1

(5.4) Revisions to technical documents are:

(a) Recorded as to date, serial number, etc. when the revision becomes effective. 5

(b) Recorded as to the date the revision was made on written specifications. 3

(c) Not recorded with any degree of precision. 1

A.5 DOCUMENTATION CONTROL (continued)

SCORE

(5.5) Procedures for making revisions to computer software programs are:

(a) Clearly spelled out in written form with the line of authority indicated. 5

(b) Not recorded but changes must be approved by the present supervisor/manager. 3

(c) Not recorded and left to the discretion of the programmer. 1

(5.6) In revising software program documentation, the revisions are:

(a) Clearly spelled out in written form, with reasons for the change and the authority for making the change distributed to all parties affected by the change. 5

(b) Incorporated by the programmer and communicated through memoranda to key people. 3

(c) Incorporated by the programmer at his will. 1

(5.7) Changes to software program documentation are:

(a) Analyzed to make sure that any harmful side effects are known and controlled prior to revision effectivity. 5

(b) Incorporated on a trial basis, monitoring the results to see if the revision has a net beneficial effect. 3

(c) Incorporated immediately with action for detecting and correcting side effects taken as necessary. 1

A.5 DOCUMENTATION CONTROL (continued)

SCORE

(5.8) Revisions to software program documentation are:

(a) Recorded as to date, program name or number, etc., when the revision becomes effective. 5

(b) Recorded as to the date the revision was made. 3

(c) Not recorded with any degree of precision. 1

A.6 PREVENTIVE MAINTENANCE

(6.1) Preventive maintenance procedures are:

(a) Clearly defined and written for all measurement systems and support equipment. 5

(b) Clearly defined and written for most of the measurement systems and support equipment. 3

(c) Defined and written for only a small fraction of the total number of systems. 1

(6.2) Preventive maintenance activities are documented:

(a) On standard forms in station log books. 5

(b) Operator/analyst summary in log book. 3

(c) As operator/analyst notes. 1

(6.3) Preventive maintenance procedures as written appear adequate to insure proper equipment operation for:

(a) All measurement systems and support equipment. 5

(b) Most of the measurement systems and support equipment. 3

(c) Less than half of the measurement systems and support equipment. 1

A.6 PREVENTIVE MAINTENANCE

SCORE

(6.4) A review of the preventive maintenance records indicates that:

(a) Preventive maintenance procedures have been carried out on schedule and completely documented. 5

(b) The procedures were carried out on schedule but not completely documented. 3

(c) The procedures were not carried out on schedule all the time and not always documented. 1

(6.5) Preventive maintenance records (histories) are:

(a) Utilized in revising maintenance schedules, developing an optimum parts/reagents inventory and development of scheduled replacements to minimize wear-out failures. 5

(b) Utilized when specific questions arise and for estimating future work loads. 3

(c) Utilized only when unusual problems occur. 1

A.7 DATA VALIDATION PROCEDURES

(7.1) Data validation procedures are:

(a) Clearly defined in writing for all measurement systems. 5

(b) Defined in writing for some measurement systems, some dependent on experience, memory, and/or verbal communication. 3

(c) Only defined by experience and verbal communication. 1

A.7 DATA VALIDATION PROCEDURES (continued)

SCORE

(7.2) Data validation procedures are:

(a) A coordinated combination of computerized and manual checks applied at different levels in the measurement process. 5

(b) Applied with a degree of completeness at no more than two levels of the measurement process. 3

(c) Applied at only one level of the measurement process. 1

(7.3) Data validation criteria are documented and include:

(a) Limits on: (1) operational parameters such as flow rates; (2) calibration data, (3) special checks unique to each measurement; e.g., successive values/averages; (4) statistical tests; e.g., outliers; (5) manual checks such as hand calculations. 5

(b) Limits on the above type checks for most of the measurement systems. 3

(c) Limits on some of the above type checks for only the high-priority measurements. 1

(7.4) Acceptable limits as set are reasonable and adequate to insure the detection of invalid data with a high probability for:

(a) All measurement systems. 5

(b) At least 3/4 of the measurement systems. 3

(c) No more than 1/2 of the measurement systems. 1

A.7 DATA VALIDATION PROCEDURES (continued)

SCORE

(7.5) Data validation activities are:

(a) Recorded on standard forms at all levels of the measurement process. 5

(b) Recorded in the operator's/analyst's log book. 3

(c) Not recorded in any prescribed manner. 1

(7.6) Examination of data validation records indicates that:

(a) Data validation activities have been carried out as specified and completely documented. 5

(b) Data validation activities appear to have been performed but not completely documented. 3

(c) Data validation activities, if performed, are not formally documented. 1

(7.7) Data validation summaries are:

(a) Prepared at each level or critical point in the measurement process and forwarded to the next level with the applicable block of data. 5

(b) Prepared by and retained at each level. 3

(c) Not prepared at each level nor communicated between levels. 1

(7.8) Procedures for deleting invalidated data are:

(a) Clearly defined in writing for all levels of the measurement process, and invalid data are automatically deleted when one of the computerized validation criteria is exceeded. 5

(b) Programmed for automatic deletion when computerized validation criteria are exceeded but procedures not defined when manual checks detect invalid data. 3

(c) Not defined for all levels of the measurement process. 1

A.7 DATA VALIDATION PROCEDURES (continued)

SCORE

(7.9) Quality audits (i.e., both on-site system reviews and/or quantitative performance audits) independent of the normal operations are:

(a) Performed on a random but regular basis to ensure and quantify data quality. 5

(b) Performed whenever a suspicion arises that there are areas of ineffective performance. 3

(c) Never performed. 1

A.8 PROCUREMENT AND INVENTORY PROCEDURES

(8.1) Purchasing guidelines are established and documented for:

(a) All equipment and reagents having an effect on data quality. 5

(b) Major items of equipment and critical reagents. 3

(c) A very few items of equipment and reagents. 1

(8.2) Performance specifications are:

(a) Documented for all items of equipment which have an effect on data quality. 5

(b) Documented for the most critical items only. 3

(c) Taken from the presently used items of equipment. 1

(8.3) Reagents and chemicals (critical items) are:

(a) Procured from suppliers who must submit samples for test and approval prior to initial shipment. 5

(b) Procured from suppliers who certify they can meet all applicable specifications. 3

(c) Procured from suppliers on the basis of price and delivery only. 1

A.8 PROCUREMENT AND INVENTORY PROCEDURES (continued)

SCORE

(8.4) Acceptance testing for incoming equipment is:

(a) An established and documented inspection procedure to determine if procurements meet the quality assurance and acceptance requirements. Results are documented. 5

(b) A series of undocumented performance tests performed by the operator before using the equipment. 3

(c) The receiving document is signed by the responsible individual indicating either acceptance or rejection. 1

(8.5) Reagents and chemicals are:

(a) Checked 100% against specification, quantity, and for certification where required and accepted only if they conform to all specifications. 5

(b) Spot-checked for proper quantity and for shipping damage. 3

(c) Released to analyst by the receiving clerk without being checked as above. 1

(8.6) Information on discrepant purchased materials is:

(a) Transmitted to the supplier with a request for corrective action. 5

(b) Filed for future use. 3

(c) Not maintained. 1

(8.7) Discrepant purchased materials are:

(a) Submitted to a review by Quality Control and Chief Chemist for disposition. 5

(b) Submitted to Service Section for determination on acceptability. 3

(c) Used because of scheduling requirements. 1

A.8 PROCUREMENT AND INVENTORY PROCEDURES (continued)

SCORE

(8.8) Inventories are maintained on:

(a) First-in, first-out basis. 5

(b) Random selection in stock room. 3

(c) Last-in, first-out basis. 1

(8.9) Receiving of materials is:

(a) Documented in a receiving record log, giving a description of the material, the date of receipt, results of acceptance test, and the signature of the responsible individual. 5

(b) Documented in a receiving record log with material title, receipt date, and initials of the individual logging the material in. 3

(c) Documented by filing a signed copy of the requisition. 1

(8.10) Inventories are:

(a) Identified as to type, age, and acceptance status. 5

(b) Identified as to material only. 3

(c) Not identified in writing. 1

(8.11) Reagents and chemicals which have limited shelf life are:

(a) Identified as to shelf life expiration data and systematically issued from stock only if they are still within that date. 5

(b) Issued on a first-in, first-out basis. expecting that there is enough safety factor so that the expiration date is rarely exceeded. 3

(c) Issued at random from stock. 1

A.9 PERSONNEL TRAINING PROCEDURES

SCORE

(9.1) Training of new employees is accomplished by:

(a) A programmed system of training where elements of training, including quality standards, are included in a training checklist. The employee's work is immediately rechecked by supervisors for errors or defects and the information is fed back instantaneously for corrective action. 5

(b) On-the-job training by the supervisor who gives an overview of quality standards. Details of quality standards are learned as normal results are fed back to the chemist. 3

(c) On-the-job learning with training on the rudiments of the job by senior coworkers. 1

(9.2) When key personnel changes occur:

(a) Specialized knowledge and skills are retained in the form of documented methods and descriptions. 5

(b) Replacement people can acquire the knowledge of their predecessors from coworkers, supervisors, and detailed study of the specifications and memoranda. 3

(c) Knowledge is lost and must be regained through long experience or trial-and-error. 1

(9.3) The people who have an impact on quality, e.g., calibration personnel, maintenance personnel, bench chemists, supervisors, etc., are:

(a) Trained in the reasons for and the benefits of standards of quality and the methods by which high quality can be achieved. 5

(b) Told about quality only when their work falls below acceptable levels. 3

(c) Are reprimanded when quality deficiencies are directly traceable to their work. 1

A.9 PERSONNEL TRAINING PROCEDURES (continued)

SCORE

(9.4) The employee's history of training accomplishments is maintained through:

(a) A written record maintained and periodically reviewed by the supervisor. 5

(b) A written record maintained by the employee. 3

(c) The memory of the supervisor/employee. 1

(9.5) Employee proficiency is evaluated on a continuing basis by:

(a) Periodic testing in some planned manner with the results of such tests recorded. 5

(b) Testing when felt necessary by the supervisor. 3

(c) Observation of performance by the supervisor. 1

(9.6) Results of employee proficiency tests are:

(a) Used by management to establish the need for and type of special training. 5

(b) Used by the employee for self-evaluation of needs. 3

(c) Used mostly during salary reviews. 1

A.10 FEEDBACK AND CORRECTIVE ACTION

(10.1) A and corrective action mechanism to assure that problems are reported to those who can correct them and that a closed loop mechanism is established to assure that appropriate corrective actions have been taken is:

(a) Clearly defined in writing with individuals assigned specific areas of responsibility. 5

(b) Written in general terms with no assignment of responsibilities. 3

(c) Not formalized but left to the present supervisors/managers. 1

A.10 FEEDBACK AND CORRECTIVE ACTION (continued)

SCORE

(10.2) Feedback and corrective action activities are:

(a) Documented on standard forms. 5

(b) Documented in the station log book. 3

(c) Documented in the operator's/analyst's notebook. 1

(10.3) A review of corrective action records indicates that:

(a) Corrective actions were systematic, timely, and fully documented. 5

(b) Corrective actions were not always systematic, timely, or fully documented. 3

(c) A closed loop mechanism did not exist. 1

(10.4) Periodic summary reports on the status of corrective action are distributed by the responsible individual to:

(a) All levels of management. 5

(b) One level of management only. 3

(c) The group generating the report only. 1

(10.5) The reports include:

(a) A listing of major problems for the reporting period; names of persons responsible for corrective actions; criticality of problems; due dates; present status; trend of quality performance (i.e., response time, etc.); listing of items still open from previous reports. 5

(b) Most of the above items. 3

(c) Present status of problems and corrective actions. 1

A.11 CALIBRATION PROCEDURES

SCORE

(11.1) Calibration procedures are:

(a) Clearly defined and written out in step-by-step fashion for each measurement system and support device. 5

(b) Defined and summarized for each system and device. 3

(c) Defined but operational procedures developed by the individual. 1

(11.2) Calibration procedures as written are:

(a) Judged to be technically sound and consistent with data quality requirements. 5

(b) Technically sound but lacking in detail. 3

(c) Technically questionable and lacking in detail. 1

(11.3) Calibration standards are:

(a) Specified for all systems and measurement devices with written procedures for assuring, on a continuing basis, traceability to primary standards. 5

(b) Specified for all major systems with written procedures for assuring traceability to primary standards. 3

(c) Specified for all major systems but no procedures for assuring traceability to primary standards. 1

(11.4) Calibration standards and traceability procedures as specified and written are:

(a) Judged to be technically sound and consistent with data quality requirements. 5

(b) Standards are satisfactory but traceability is not verified frequently enough. 3

(c) Standards are questionable. 1

A.11 CALIBRATION PROCEDURES (continued)

SCORE

(11.5) Frequency of calibration is:

(a) Established and documented for each measurement system and support measurement device. 5

(b) Established and documented for each major measurement system. 3

(c) Established and documented for only certain measurement systems. 1

(11.6) A review of calibration data indicates that the frequency of calibration as implemented:

(a) Is adequate and consistent with data quality requirements. 5

(b) Results in limits being exceeded a small fraction of the time. 3

(c) Results in limits being exceeded frequently. 1

(11.7) A review of calibration history indicates that:

(a) Calibration schedules are adhered to and results fully documented. 5

(b) Schedules are adhered to most of the time. 3

(c) Schedules are frequently not adhered to. 1

(11.8) A review of calibration history and data validation records indicates that:

(a) Data are always invalidated and deleted when calibration criteria are exceeded. 5

(b) Data are not always invalidated and/or deleted when criteria are exceeded. 3

(c) Data are frequently not invalidated and/or deleted when criteria are exceeded. 1

A.11 CALIBRATION PROCEDURES (continued)

SCORE

(11.9) Acceptability requirements for calibration results are:

(a) Defined for each system and/or device requiring calibration including elapsed time since the last calibration as well as maximum allowable change from the previous calibration. 5

(b) Defined for all major measurement systems. 3

(c) Defined for some major measurements systems only. 1

(11.10) Acceptability requirements for calibration results as written are:

(a) Adequate and consistent with data quality requirements: 5

(b) Adequate but others should be added. 3

(c) Inadequate to ensure data of acceptable quality. 1

(11.11) Calibration records (histories) are:

(a) Utilized in revising calibration schedules (i.e., frequency). 5

(b) Utilized when specific questions arise and reviewed periodically for trends, completeness, etc. 3

(c) Utilized only when unusual problems occur. 1

A.12 FACILITIES/EQUIPMENT

(12.1) Facilities/Equipment are:

(a) Adequate to obtain acceptable results. 5

(b) Adequate to obtain acceptable results most of the time. 3

(c) Additional facilities and space are needed. 1

A.12 FACILITIES/EQUIPMENT (continued)

SCORE

(12.2) Facilities, equipment, and materials are:

(a) As specified in appropriate documentation and/or standards. 5

(b) Generally as specified in appropriate standards. 3

(c) Frequently different from specifications. 1

(12.3) Housekeeping reflects an orderly, neat, and effective attitude of attention to detail in:

(a) All of the facilities. 5

(b) Most of the facilities. 3

(c) Some of the facilities. 1

(12.4) Maintenance Manuals are:

(a) Complete and readily accessible to maintenance personnel for all systems, components, and devices. 5

(b) Complete and readily accessible to maintenance personnel for all major systems, components, and devices. 3

(c) Complete and accessible for only a few of the systems. 1

A.13 RELIABILITY

(13.1) Procedures for reliability data collection, processing, and reporting are:

(a) Clearly defined and written for all system components. 5

(b) Clearly defined and written for major components of the system. 3

(c) Not defined. 1

A.13 RELIABILITY (continued)

SCORE

(13.2) Reliability data are:

(a) Recorded on standard forms. 5

(b) Recorded as operator/analyst notes. 3

(c) Not recorded. 1

(13.3) Reliability data are:

(a) Utilized in revising maintenance and/or replacement schedules. 5

(b) Utilized to determine optimum parts inventory. 3

(c) Not utilized in any organized fashion. 1

APPENDIX B

STANDARD TECHNIQUES USED IN QUANTITATIVE PERFORMANCE AUDITS

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Ambient air techniques[a]

Pollutant	EPA method or number	Bias (absolute, or percent of mean concentration)	Precision (absolute, or coefficient of variation)		Comments	Reference
			Within-laboratory	Between-laboratory		
SO_2	6	0	5-13 $\mu g/m^3$, from x = 0-1000 $\mu g/m^3$	10-25 $\mu g/m^3$ from x = 0-1000 $\mu g/m^3$	Lower limit of detection is 25 $\mu g/m^3$. Flow rate changes, sampling train leakage are primary error sources.	EPA-R4-73-028d
NO, NO_2 NO_x	Chemiluminescent	0	7-8% at 100 $\mu g/m^3$ (0.05 ppm)	-	Lower limit of detection is 10 $\mu g/m^3$ (0.005 ppm). Errors are associated with calibration and instrument drift (from zero and span settings).	d
Photochemical oxidants	Chemiluminescent	-35 to -15%[c] from 0.05 to 0.50 ppm	0.0033 + 0.0255 × (0-0.5 ppm)[c]	0.0008 + 0.0355 × (0-0.5 ppm) -0.0051 + 0.0690 × (0.15-0.5 ppm)	Lower detection limit is 0.0065 ppm	EPA-R4-73-028c
CO	NDIR	+2.5	0.6 mg/m^3	0.8 - 1.6 mg/m^3 (nonlinear variation) over 0-60 mg/m^3	Lower detection limit is 0.3 mg/m^3. Interference of water vapor is significant.	EPA-R4-73-028a

Ambient air techniques (con.)

Pollutant	EPA method or number	Bias (absolute, or percent of mean concentration)	Precision (absolute, or coefficient of variation)		Comments	Reference
			Within-laboratory	Between-laboratory		
Particulates	High-Volume	No information	3%	3.7%	Minimum detectable limit is 3 mg. Shorter sampling periods give less precise results, biased high.	EPA-R4-73-028b
NO_2	Arsenite	-3% (50-300 $\mu g/m^3$)	8 $\mu g/m^3$ (50-300 $\mu g/m^3$)	11 $\mu g/m^3$ (50-300 $\mu g/m^3$)	A tentative method. Lower detectable limit is 9 $\mu g/m^3$.	EPA-R4-73-280o

[a]This table is a summary of information contained in the cited references, all of which are quality assurance guideline manuals published by EPA. Collaborative test results are cited, if available, in the manuals.

[b]x = pollutant concentration.

[c]EPA-650/4-75/016.

[d]Guidelines for Development of a Quality Assurance Program for the Continuous Measurement of Nitrogen Dioxide in the Ambient Air (Chemiluminescent), Smith & Nelson, Research Triangle Institute, Research Triangle Park, N.C. 27709.

Source Sampling Techniques[a]

Pollutant	EPA method or number	Bias (absolute, or percent of mean concentration)	Precision (absolute, or coefficient of variation)		Comments	Reference
			Within-laboratory	Between-laboratory		
SO_2	6	0	3.9	5.5	Major error source is difficulty of obtaining reproducible titration endpoints. Minimum detectable limit is 3 ppm.	EPA-650/14-74-005-e
SO_2 and SO_3/H_2SO_4	8	-2% (analysis only)	0.1 g/m^3	0.11 g/m^3	Same analysis technique as Method 6 above.	EPA-650/14-74-005-g
		-2% (analysis only)	60%	65%		
NO_x	7	0	7%	10%	Grab sample; largest error source is failure to recalibrate spectrophotometer.	EPA-650/14-74-005-f
CO	10	+7 ppm	13 ppm	25 ppm	Analyzer drift and CO_2 interference are largest problems. Minimum detectable limit is 20 ppm.	EPA-650/14-74-005-h
Particulates	5	No information	10-30%	20-40%	Numerous small error sources associated with stack sampling.	EPA-650/14-74-005-d
Visible emissions	9	+1.4% opacity	2% opacity	2.5%	Good results depend to a great extent on the effective training of observers.	EPA-650/14-74-005-i
Be	104	-20%, average	44%	58%		EPA-650/14-74-005-k

[a]This table is a summary of information contained in the cited references, all of which are quality assurance guidelines manuals.

APPENDIX C

COMPARISON OF ANALYSES OF LIMESTONE SLURRY

APPENDIX C COMPARISON OF ANALYSIS OF LIMESTONE SLURRY

Cooperating laboratories were:

1. TVA Power Service Center Laboratory, Chattanooga, Tennessee - Mr. John Rose, contact
2. TVA Power Service Center Laboratory, Muscle Shoals, Alabama - Dr. Guerry McClellon, contact
3. Research Triangle Institute, Research Triangle Park, North Carolina - Dr. D. E. Wagoner, contact

Results from RTI laboratories are presented in two sections. One set of data was obtained on slurry which was filtered at the Shawnee Laboratory. The second set of data results from analysis of samples filtered in the RTI laboratory.

The first eight matrixes present results for each element: calcium, magnesium, and total sulfur in the solid; and calcium, magnesium, sodium, potassium, and chloride in the liquid. The next five matrixes give results of all analyses for each laboratory, with Shawnee results listed first. The last four matrixes break down the total sulfur and calcium analyses into results by a standard "wet" technique, by X-ray fluorescence using Shawnee standard values, and by X-ray fluorescence using RTI-derived standard values on the Shawnee standard material.

Table 1. Analysis for calcium in slurry solid

Sample: Ca (as CaO) Wt % / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	24.73	24.50	22.78	21.05	22.87
RTI (Shawnee filtered)	22.12	19.03	19.60	18.40	19.46
RTI (RTI filtered)	17.99	17.96	18.63	18.13	19.32
Chattanooga (TVA)	23.45	23.24	23.02	22.46	22.46
Muscle Shoals (TVA)	23.6	23.1	23.6	22.6	22.9

Table 2. Analysis for magnesium in slurry solid

Sample: Mg (as MgO) Wt % / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	0.30	0.29	0.28	0.27	0.29
RTI (Shawnee filtered)	0.43	0.48	0.39	0.36	0.42
RTI (RTI filtered)	0.61	0.52	0.34	0.39	0.35
Chattanooga (TVA)	0.65	0.56	0.56	0.61	0.61
Muscle Shoals (TVA)	0.25	0.25	0.24	0.25	0.24

Table 3. Analysis for total sulfur in slurry solid

Sample: TS (as SO_3) wt % / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	34.78	34.16	31.22	28.17	31.64
RTI (Shawnee filtered)	36.70	32.00	33.03	30.53	32.80
RTI (RTI filtered)	28.60	27.00	31.13	28.78	32.03
Chattanooga (TVA)	30.5	30.7	30.0	29.2	29.6
Muscle Shoals (TVA)	31.3	30.9	31.4	29.7	30.4

Table 4. Analysis for calcium in slurry filtrate

Sample: Ca (ppm) / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	1720	1710	1810	2090	2315
RTI (Shawnee filtered)	1775	1825	1810	1708	1885
RTI (RTI filtered)	1700	1810	1730	1720	1825
Chattanooga (TVA)	1756	1740	1676	1596	1732
Muscle Shoals (TVA)	1787	1787	1716	1787	1716

Table 5. Analysis for magnesium in slurry filtrate

Sample: Mg (ppm) / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	733	699	662	691	698
RTI (Shawnee filtered)	785	945	813	1000	1115
RTI (RTI filtered)	730	805	805	795	770
Chattanooga (TVA)	768	734	763	780	816
Muscle Shoals (TVA)	724	724	724	724	784

Table 6. Analysis for sodium in slurry filtrate

Sample: Na (ppm) / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	71	57	70	73	82
RTI (Shawnee filtered)	71	69	79	75	77
RTI (RTI filtered)	153	161	180	176	145
Chattanooga (TVA)	66	62	64	66	69
Muscle Shoals (TVA)	41	37	37	41	41

Table 7. Analysis for potassium in slurry filtrate

Sample: K / Laboratory	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Shawnee	118	121	123	126	153
RTI (Shawnee filtered)	103	101	105	108	111
RTI (RTI filtered)	116	102	114	118	116
Chattanooga (TVA)	107	96	107	116	116
Muscle Shoals (TVA)	58	54	50	58	58

Table 8. Analysis for chloride in slurry filtrate

Sample: Cl (ppm) / Laboratory	11/18/75 1100	11/18/75 1500	11/18/75 2300	11/19/75 1100	11/19/75 2300
Shawnee	3651	3580	3545	3545	3580
RTI (Shawnee filtered)	3697	3700	3855	3660	3987
RTI (RTI filtered)	3621	3638	3754	3519	3566
Chattanooga (TVA)	3692	3543	3571	3571	3628
Muscle Shoals (TVA)	3800	3700	3600	3600	3700

Table 9. Laboratoty: Shawnee (TVA)

SOLID (Wt.%)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Ca (CaO)	24.73	19.03	22.78	21.05	22.87
Mg (MgO)	0.30	0.48	0.28	0.27	.29
TS (SO_3)	34.78	32.00	31.22	28.17	31.64
LIQUID (ppm)					
Ca	1720	1710	1810	2090	2315
Mg	733	699	662	691	698
Na	71	57	70	73	82
K	118	121	123	126	153
Cl	3651	3580	3545	3545	3580

Table 10. Laboratory: RTI (Shawnee filtered)

SOLID (Wt. %)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Ca (CaO)	22.12	19.03	19.10	18.41	19.46
Mg (MgO)	0.43	0.48	0.39	0.36	0.42
TS (SO_3)	36.7	32.00	33.03	30.53	32.80
LIQUID (ppm)					
Ca	1775	1825	1810	1708	1885
Mg	785	945	913	1000	1115
Na	71	69	79	75	77
K	103	101	105	108	111
Cl	3697	3700	3855	3660	3987

Table 11. Laboratory: RTI

SOLID (Wt. %)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Ca (CaO)	17.99	19.96	18.63	18.13	19.32
Mg (MgO)	0.61	0.52	0.34	0.39	0.35
TS (SO_3)	28.6	27.0	31.13	28.78	32.03
LIQUID (ppm)					
Ca	1700	1810	1730	1720	1825
Mg	730	805	805	795	770
Na	153	101	180	176	145
K	116	102	114	118	116
Cl	3621	3638	3754	3519	3566

Table 12. Laboratory: Muscle shoals (TVA)

SOLID (Wt. %)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Ca (CaO)	23.6	23.1	23.6	22.6	22.9
Mg (MgO)	0.25	0.25	0.24	0.25	0.24
TS (SO_3)	31.3	30.9	31.4	29.7	36.4
LIQUID (ppm)					
Ca	1787	1787	1716	1787	1716
Mg	724	724	724	724	784
Na	41	37	37	41	41
K	58	54	50	58	58
Cl	3800	3700	3600	3600	3700

Table 13. Laboratory: Chattanooga (TVA)

SOLID (Wt. %)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
Ca (CaO)	23.45	23.24	23.02	22.46	22.46
Mg (MgO)	0.65	0.56	0.56	0.61	0.61
TS (SO_3)	30.5	30.7	30.0	29.2	29.6
LIQUID (ppm)					
Ca	1756	1740	1676	1596	1732
Mg	768	734	763	780	816
Na	66	62	64	66	67
K	107	96	107	116	116
Cl	3692	3543	3571	3571	3628

Table 14. Total sulfur determinations, Shawnee filtered samples analyzed at RTI

wt % (as SO_3)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
By $BaCl_2$ precipitation	36.7	32.00	33.03	30.53	32.80
By X-Ray fluorescence					
Using Shawnee X-Ray standard number for TS	22.08*	21.25	23.15	27.75	22.95
Using RTI determined number for TS					
Shawnee X-Ray Standard (as SO_3)*					
Shawnee given	28.45				
RTI determined	28.38				

* Shawnee and RTI TS determinations on the XRF standard were virtually identical, so the Shawnee TS value only was used in calculating wt % TS in each sample.

Table 15. Total sulfur determinations, RTI filtered and analyzed samples

	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
$BaCl_2$ precipitation	28.6	27.00	31.13	28.78	32.03
X-Ray fluorescence					
Using Shawnee X-Ray standard number for TS	35.13	30.28	29.8	34.45	29.18
Using RTI-determined number for TS					

Table 16. Calcium determinations, Shawnee filtered samples analyzed at RTI

wt % (as CaO)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
By AA	22.12	19.03	19.60	18.41	19.46
By X-Ray fluorescence					
Using Shawnee X-Ray standard number for CaO	23.13	25.41	22.25	25.76	22.89
Using RTI determined number for CaO	20.09	22.06	19.32	22.37	19.88
Shawnee X-Ray Standard (as CaO)					
Shawnee given	25.41				
RTI determined	22.06				

Table 17. Calcium determinations, RTI filtered and analyzed samples

wt % (as CaO)	11/18/75			11/19/75	
	1100	1500	2300	1100	2300
By AA	17.99	17.96	18.63	18.13	19.32
By X-Ray fluorescence					
Using Shawnee X-Ray standard number for CaO	28.35	25.52	26.17	27.68	26.32
Using RTI-determined number for CaO	24.61	22.16	22.72	24.04	22.85

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15. SUPPLEMENTARY NOTES Project officer for this report is L. D. Johnson, Mail Drop 62, Ext 2557.

16. ABSTRACT The report outlines results of a project: to develop a set of quality assurance guidelines for EPA demonstration projects; to implement a short-term quality assurance program at the EPA wet limestone scrubber facility at the Shawnee steam/electric plant; and to modify the guidelines in light of the Shawnee operating experience. The set of quality assurance guidelines and detailed results of the Shawnee program are included in two other reports prepared during the project.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group	
Air Pollution	Industrial Processes	Air Pollution Control	13B	
Quality Assurance	Instruments	Stationary Sources	13H,14D	14B
Quality Control	Sulfur Dioxide	Field Application		07B
Scrubbers	Dust	Control Laboratory	07A	11G
Limestone	Sampling	Particulate	08G	
Flue Gases	Weight Measurement		21B	

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